Approved for Release: 2015/01/05 C00037492

[redacted]

(22

ANALYTICAL RESEARCH DIVISION
RESEARCH DIRECTORATE

21 August 1984

Analysis/Evaluation of Brown Powders

A shipment designated 10027ZZZ also identified with TH830513-37DL was received by the Analytical Research Division 3 June 1983 from FSTC. The shipment consisted of one light brown powder and one dark brown powder, each containerized in individual rubber septum capped glass vials, fig. 1. The vials were containerized in a plastic bag [redacted] The light brown powder was designated 10027ZZZ-1 and the dark brown powder was designated 10027ZZZ-2. b3

Vapor samples withdrawn from within each vial were subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of each powder was extracted with chloroform. Another portion of each powder was extracted with 1:1 methanol:water. The solvent extracts were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC) and infrared spectrometry (IR). The neat powders were also evaluated by scanning electron microscopy (SEM).

10027ZZZ-1 light brown powder

The GC/MS analysis of the vapor associated with the powder contained no definitive mass spectra. The GC/MS spectra of the chloroform solubles identified the presence of trinitrotoluene (TNT), hexamethylene tetramine, aliphatic hydrocarbons from C_{23} to C_{26} and a base peak M/z = 75, 116, 179, 168, 179 which could be nitronaphthalene. Ion chromatography separated and quantitated the presence of 1 ppm fluoride and a possible trace of cyanide. TLC separated a UV absorber at Rf 0.8. Reaction of the developed TLC plate for alkylating agents gave a purple product at Rf 0.4 and 0.8. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.- IR analysis identified the presence of aliphatic hydrocarbons and aromatic nitrates, primarily TNT. SEM evaluation showed the neat material was a granular, partially crystalline solid.

10027ZZZ-2 dark brown solid

The GC/MS spectra of the vapor associated with the powder identified the presence of diethyleneglycol mono ethylether and 2,6,-di-t-butyl-4-ethenylphenol as the major components and benzene, benzaldehyde, toluene, xylene, nonanol and 2,4-di-t-buty-4-ethylphenol as minor components. The GC/MS analysis of the chloroform solubles identified the major component as TNT and dinitrotoluene the minor component. Ion chromatography separated and quantitated 0.5 ppm fluoride and a possible trace of cyanide. TLC separated one component, a UV absorber at Rf 0.60-0.82 which reacted with p-anisaldehyde reagent to give a beige color. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. The IR spectra identified the presence of aromatic nitrates, primarily TNT. SEM evaluation showed the neat solid was a granular, partially crystalline material, nearly identical to the 10027ZZZ-1 sample.

[redacted]

Classified by: CIA [redacted]
Declassify: OADR

Approved for Release: 2015/01/05 C00037492

ENCL 7

Approved for Release: 2015/01/05 C00037492

[redacted]

Conclusions:

Both powders were determined to be primarily TNT. The lighter colored powder, 10027ZZZ-1, appeared to be relatively pure. The darker powder, 10027ZZZ-2, appeared to contain solvents and manufacturing impurities.

[redacted]

Approved for Release: 2015/01/05 C00037492

Approved for Release: 2015/01/05 C00037492

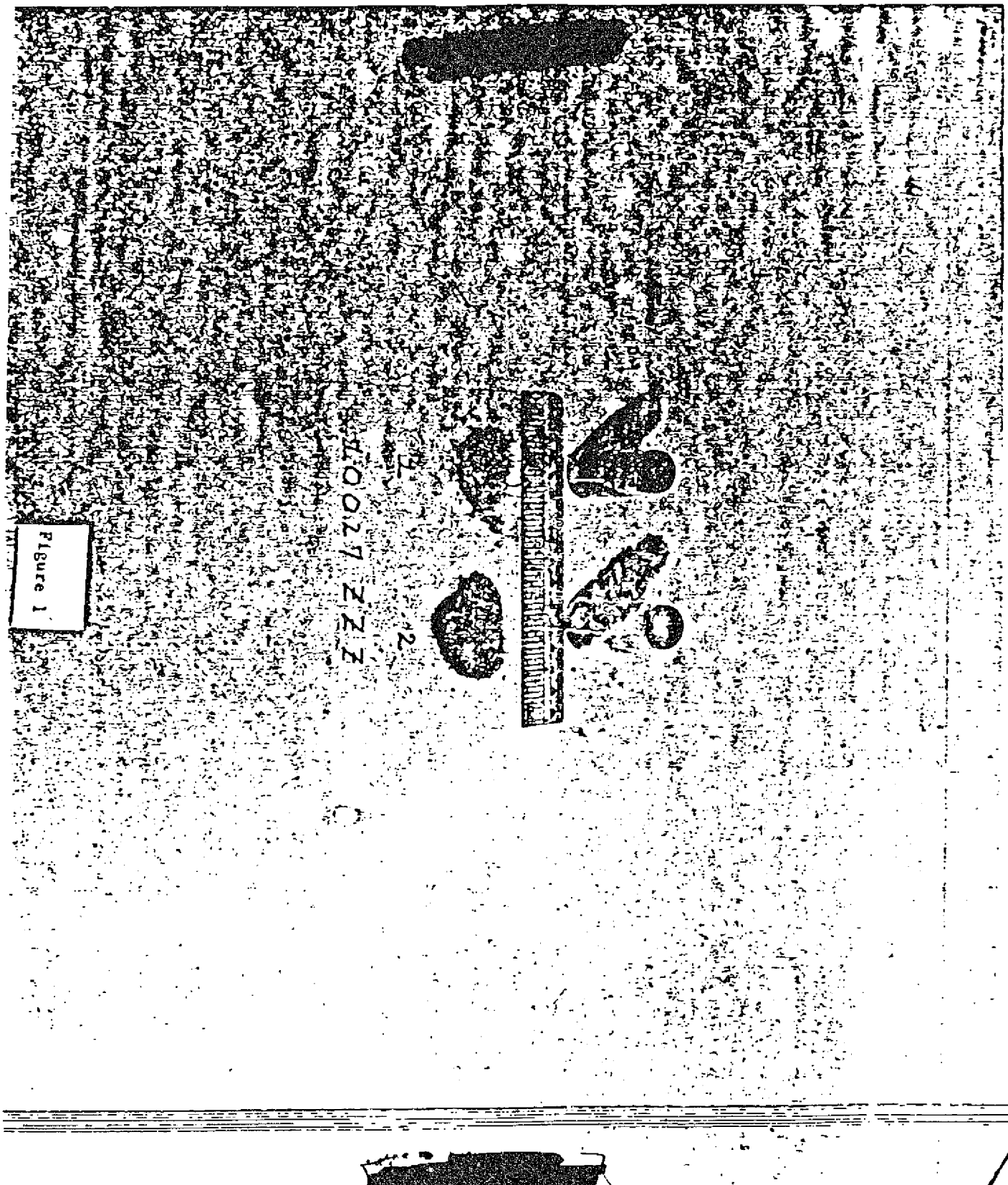

Figure 1

Approved for Release: 2015/01/05 C00037492